

# REPORT DOCUMENTATION PAGE

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13. ABSTRACT (Maximum 200 words)  In this reporting period, processing of nanostructured powders, consolidation of nanostructured powders, computer simulation, microstructural characterization, and mechanical deformation tests were carried out. The nanostructured powders and bulk nanostructured materials were produced successfully via optimization of processing parameters. The observed highly grain stability can be explained by a grain boundary pinning mechanism arising from the dispersoid particles as well as impurity segregation. The microstructures of the as-extruded and the deformed bulk nanocrystalline aluminum alloys were investigated using transmission electron microscopy. A nanoindentation technique and traditional mechanical tests as well as computer simulation were also used to evaluate the mechanical performance. A discrete dislocation dynamics model has been developed to bridge the gap between atomic simulations and continuum approaches. It has the potential to provide a rigorous description of the complex relationships between the macroscopic mechanical behavior of materials and the underlying fundamental physical mechanisms. The tensile test results showed that grain size effects, solid solution strengthening, Orowan strengthening and dislocation strengthening contribute significantly to the properties of the cryomilled Al alloys. The future work for the next period is also discussed in the report.					
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**1. List of Publications**

- (1) B. Q. Han, Z. Lee, S. R. Nutt, E. J. Lavernia, and F. A. Mohamed, "Mechanical Properties of An Ultrafine-Grained Al-7.5%Mg Alloy," *Metall. Mater. Trans. A*, in press, 2002.
- (2) F. Zhou, R. Luck, K. Lu, E.J. Lavernia and M. Ruhle, "Amorphous to crystalline transformation induced by thermal annealing of a metastable Al90Fe10 composite", *Philosophical Magazine A*, Vol. 82, 1003-1015, 2002.
- (3) L. Ajdelsztajn, J. Lee, K. Chung, F.L. Bastian and E.J. Lavernia, "Synthesis and Nanoindentation Study of HVOF Thermal Sprayed Nanocrystalline and Near-Nanocrystalline Ni Coatings", *Metallurgical and Materials Transactions, Vol. 33A*, pp. 647-65, 2002.
- (4) J. He and E.J. Lavernia, "Development of Nanocrystalline Structure during Cryomilling of Inconel 625," *Journal of Materials Research, Vol. 16*, 2724-2732, 2001.
- (5) R.W. Hayes, R. Rodriguez and E.J. Lavernia, "The mechanical behavior of a cryomilled Al-10Ti-2Cu alloy" *Acta Materialia*, vol. 49, 4055-4068, 2001.
- (6) J. Lee, F. Zhou, K.H. Chung, N.J. Kim, and E.J. Lavernia, "Grain Growth of Nanocrystalline Ni Powders Prepared by Cryomilling", *Metallurgical and Materials Transactions*, Vol. 32A, 3109-3115, 2001.
- (7) Z. Lee, R. Rodriguez, E. J. Lavernia and S. R. Nutt, "Microstructural Evolution of Cryomilled Nanocrystalline Al-Ti-Cu Alloy," *Ultrafine Grained Materials II*; Edited by Y.T. Zhu, et al., TMS, 653-659, 2002.
- (8) F. Zhou, Y.T. Chou and E. J. Lavernia, "Mechanical Alloying in Nanostructured Fe-Zn Binary System", International Symposium on Science and Technology of Interfaces, 2002 TMS Annual Meeting, Seattle, Washington, February 17-21, 2002.
- (9) B. Q. Han, E. J. Lavernia, F. A. Mohamed, D. Matejczyk and C. Bampton, "Creep Behavior in a Cryomilled Near-Nanostructured Al-7.5%Mg Alloy," *Sixth International Conference on Nanostructured Materials*, Orlando, FL, June 16 – 21, 2002.
- (10) Z. Lee, R. Rodriguez, E. J. Lavernia and S. R. Nutt, "Deformation of Cryomilled Nanocrystalline Al-Ti-Cu and Al-Mg Alloys," *Sixth International Conference on Nanostructured Materials*, Orlando, FL, June 16 – 21, 2002.
- (11) R. Ye and E.J. Lavernia, "Numerical Study for Dislocations Emission under Indentation," *Sixth International Conference on Nanostructured Materials*, Orlando, FL, June 16 – 21, 2002.

**2. Scientific Personnel**

Dr. Leo Ajdelsztajn, post-doc (PhD degree was received in June 2002)  
Dr. Bing Q. Han, post-doc  
Dr. Riqing Ye, post-doc  
Mr. Rudy Rodriguez, PhD student  
Mr. Akshay Verma, PhD student  
Mr. Zonghoon Lee, PhD student (USC)  
Mr. Benjamin John Murray, PhD student  
Mr. Eric Menke, PhD Student

**3. Scientific Progress and Accomplishments**

The objectives of this research program are threefold: first, to establish an in-depth understanding of the factors that govern the synthesis and mechanical behavior of bulk nanostructured materials; second, to develop novel experimental and numerical technique to explore deformation evolution in bulk nanostructured materials; third, to utilize this fundamental information to develop new bulk nanostructured alloys with ultra-high strength

and good ductility. This report covers the progress made during the period from October 1, 2001 to September 30, 2002. The primary accomplishments and the future work of the next year are described as follows.

(1) Processing of nanostructured powders

Grain growth of nanocrystalline Ni powders with an average grain size of 22 nm prepared by cryogenic mechanical milling (or cryomilling) was investigated by using X-ray diffraction (XRD) and transmission electron microscopy (TEM). A dispersion of NiO and Ni<sub>3</sub>N particles with a size less than 5 nm was formed in the cryomilled powders. The Ni<sub>3</sub>N particles decomposed at 773 K. It was found that at 0.56 homologous temperature ( $T/T_M$ ), Ni grains were retained at 150 nm even after long annealing times (*e.g.*, 4 hours). For 0.45 to 0.62  $T/T_M$ , the time exponent  $n$  deduced from  $D^{1/n} - D_0^{1/n} = kt$  was 0.16 to 0.32, tending toward 0.5 as  $T/T_M$  increased. The activation energy for grain growth in the Ni sample was determined to be 113 kJ/mol, which is close to the activation energy for grain boundary self-diffusion in polycrystalline Ni. The observed highly grain stability was attributed primarily to a grain boundary pinning mechanism arising from the NiO particles as well as impurity segregation.

(2) Microstructural characterization

The microstructures of the as-extruded and the deformed bulk nanocrystalline Al-Ti-Cu and Al-Mg alloys were investigated using transmission electron microscopy and scanning electron microscopy. Grain refinement was achieved by cryomilling of elemental powders, and the cryomilled powders were then consolidated by hot isostatic pressing (HIP) and extrusion to produce bulk nanocrystalline Al-Ti-Cu and Al-Mg alloys. In an effort to enhance ductility and toughness of Al-Ti-Cu alloy, multi-scale structures were produced, including nanocrystalline grains, elongated coarse grains of pure Al, and intermediate grains (Fig. 1). Pure aluminum grains were elongated along the extrusion direction and coarse-grain bands comprising of sub-grains were formed. Nano-scale second phases were distributed along the boundaries of the intermediate grains and nanocrystalline grains. Tensile fracture tests of bulk specimens revealed the unusual failure mechanisms indicating the interactions between ductile coarse grains and nanocrystalline regions. The ductile coarse grains underwent large plastic deformation. Fractures appeared to involve nucleation and growth of voids at Al<sub>3</sub>Ti particle-aluminum grain interfaces and Al<sub>3</sub>Ti particles interfaces because of a limited plastic deformation. The coarse-grain aluminum bands enhanced the ductility and toughness of nanocrystalline aluminum alloys through crack blunting and crack bridging during fracture. In the Al-Mg alloy, nanocrystalline grains were uniform and elongated, but coarse grain bands were absent (Fig. 2). The Al-Mg alloy exhibited a different failure mechanism involving complex interactions between nanocrystalline grains.

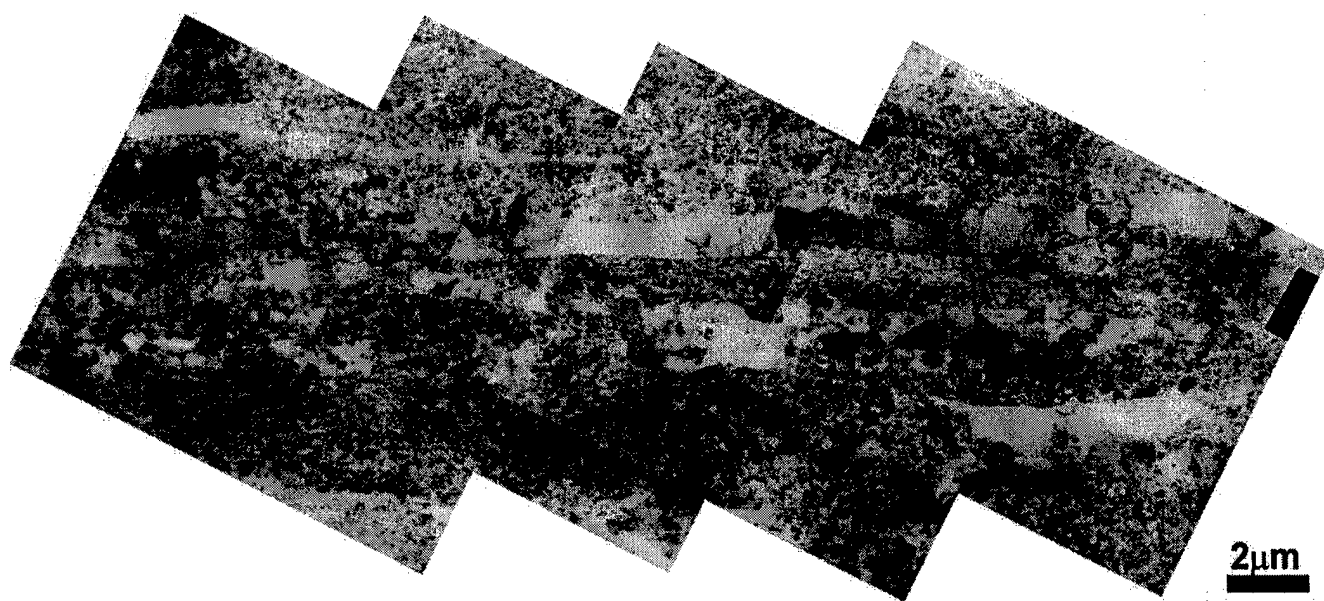


Fig. 1 Coarse-grained bands in the cryomilled Al-Ti-Cu alloy

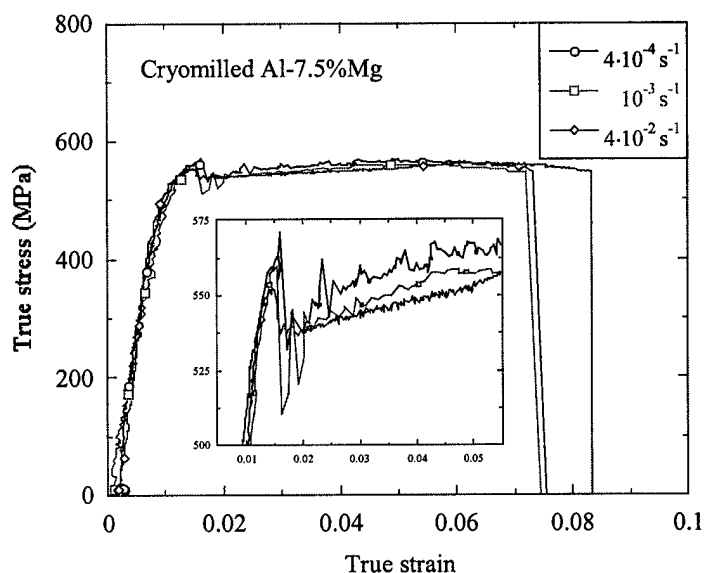


Fig. 2 Microstructure of the cryomilled Al-7.5%Mg alloy.

Fig. 3 Tensile behavior of the cryomilled Al-7.5%Mg.

### (3) Deformation mechanisms

- a The relationships between structure and properties of the cryomilled Al-7.5%Mg alloy were investigated. The microstructure of the cryomilled Al-7.5%Mg alloy consisted of equiaxed grains with an approximate size of 300 nm. Thermal treatment had only a minor effect on microstructure, as evidenced by X-ray diffraction (XRD) and transmission electron microscopy (TEM) results. Tensile behavior was characterized by high strength, high ductility and low strain hardening (Fig. 3). Tensile deformation was relatively uniform with limited necking deformation, and fracture surfaces were characterized by micro-dimples. The variation of strain rates from  $4 \cdot 10^{-4}$  to  $4 \cdot 10^{-2} \text{ s}^{-1}$  had an insignificant effect on tensile behavior. Comparison of compressive and tensile behavior revealed similar moduli and yield strengths, although post-yield behavior was markedly asymmetric. The present results indicate that grain size effects, solid solution strengthening, Orowan strengthening and dislocation strengthening contribute significantly to the properties of the cryomilled Al-7.5%Mg alloy.
- b The creep behavior of the bulk Al-7.5%Mg alloy consolidated from cryogenic mechanical milling powders was investigated at temperatures of 573, 623, and 673 K. The results show the presence of two creep regions: a low-stress region that exhibits a low stress exponent of less than 10, and a high-stress region that is characterized by a high stress exponent of approximately 35.9. An analysis of the data of the low-stress region suggested the presence of a threshold stress, which strongly depends on temperature. Creep behavior of the cryomilled Al-7.5%Mg alloy was also compared with that of coarse-grained aluminum alloys and mechanically alloying oxide-dispersion-strengthened aluminum alloys. While it is much stronger than that of coarse-grained aluminum alloys, the creep resistance of the cryomilled Al-7.5%Mg alloy is similar to that of mechanically alloying oxide-dispersion-strengthened aluminum alloys.
- c The mechanical behavior of a cryomilled Al-10Ti-2Cu (wt.%) alloy was studied by performing uniaxial tension tests at temperatures ranging from room temperature to 525°C. An elastic-nearly perfectly plastic stress-strain behavior was observed at all temperatures. Tension-compression asymmetry of the room temperature yield stress was also observed. These characteristics are in agreement with those recently reported in the literature for single-phase nanocrystalline materials. The flow stress (700 MPa at room temperature) decreases dramatically with increasing temperature. Testing of the Al-10Ti-2Cu alloy experienced by thermal exposures suggests that microstructural coarsening alone cannot account for the decrease in strength with increasing temperature. From a grain coarsening standpoint, this Al-10Ti-2Cu alloy appears to be very thermally stable. The ductility is influenced by several factors. Low levels of internal porosity along with the presence of fine oxide and carbide dispersoids contribute to lower ductility. The absence of work hardening exhibited by the Al-10Ti-2Cu also leads to reduced strain to failure. The features observed on fracture surfaces suggest that fracture occurs by the nucleation and growth of voids at particle-matrix interfaces. Evidence of fracture along prior powder particle boundaries is present as well. The microstructure consists primarily of regions containing grains measuring in

the range 30–70 nm. Large grained regions consisting of nominally pure Al ranging in size from 300 to 500 nm are also present. No evidence of dislocation activity within either the fine or large grained regions can be found in the as-extruded material. Specimens deformed at room temperature and 93 °C reveal evidence of dislocation activity within the large grain regions. Dislocation configurations suggest an Orowan bypass mechanism. No dislocations are found within the 30–70 nm size grains following tensile deformation.

(4) Computer simulation

Indentation instruments have been developed to permit measurement of elastic modulus, hardness, yield strength and other plastic properties at the nano-scale (1–100 nm). On the computational side, dislocation dynamics codes have been used in comparing the pattern of dislocations in the plastic zones beneath the indenter with experimental observations. In our study, a variation boundary integral method, in the Peierls-Nabarro framework, is used to study mechanical behavior during nano-indentation. In the simulation, framework formulated herein, the dislocations are discretized into segments that serve as the fundamental degrees of freedom. The indentation behavior consists of two parts: the first is elastic deformation, which is predicted on the basis of contact mechanics and the second, dislocation nucleation. It was found that when the applied indenter force reaches a critical load, the load-displacement curve departs from its “normal” track, which we propose is associated with the emission of dislocations. More dislocations are generated with increasing applied load. Our results suggest that a discrete dislocation dynamics model can bridge the gap between atomic simulations and continuum approaches. Moreover a discrete dislocation dynamics model has the potential to provide a rigorous description of the complex relationships between the macroscopic mechanical behavior of materials and the underlying fundamental physical mechanisms.

(5). Future work in the next year

- a Thermal stability of the nanostructured bulk pure Al: To analyze the effect of small grain size and atomic level strain on the XRD peak broadening. Also, factors affecting the stabilization of the microstructure of nanostructured bulk materials such as impurities and dispersoids will be investigated.
- b Consolidation of the cryomilled aluminum powders with different volume fraction of mixed-size nano-scale phases: Previous work showed that a small amount of coarse grains in the microstructure of bulk nanostructured materials benefit to the improvement of ductility and toughness. Bulk nanostructured aluminum alloys with different volume fractions of mixed-size grains will be manufactured in the next stage.
- c Mechanical characterization of the nanostructured aluminum alloys with different volume fraction of mixed-size nano-scale phases: Previous works showed that the ultra-high strength aluminum alloys could be achieved by consolidation of cryomilled nanostructured aluminum powders via optimization of processing parameters. On the other hand, low ductility and low fracture toughness exist in such nanostructured aluminum alloys. Therefore, it is of particular interest to maintain the high strength and high ductility as well as high toughness. The objectives of this task are: 1) to investigate the mechanical response of nanostructured materials with different volume fractions of mixed-size powders, and 2) to understand the dependence of nanosized crystalline phases on the cryomilling parameters, degassing, HIP'ing and extruding parameters, and the influence of the consolidation processing parameters on the mechanical properties. Relationship between volume fraction and distribution of mixed-size powders, mechanical properties, and processing parameters will be established.
- d Computer simulation of nanoindentation and deformation mechanisms of bulk nanostructured aluminum alloys with different volume fractions of mixed-size phases: The objective of this task is to provide a rigorous description of the complex relationships between the macroscopic mechanical behavior of nanostructured materials and the underlying fundamental physical mechanisms.